

Paint and Varnish

A TECHNICAL MAGAZINE

Vol. XXIX

JUNE, 1949

No. 6



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A TECHNICAL, EDUCATIONAL AND
PRACTICAL JOURNAL FOR THE
PRODUCTION MANAGER

Founded September, 1928

Comprising
"The Paint and Varnish Record"
Founded in 1911

Entered as Second-class matter, May, 1935, at
the Post Office at Washington, D. C.
Under the Act of March 3, 1879.

Published the First of Every Month by Paint and Varnish
PRODUCTION MANAGER

Member, Oil and Colour Chemists' Association, London, England.

M. R. DRAKE, *Publisher*
EDWARD R. DRAKE, *Editor*

Publication Office
Mills Building, Washington 6, D. C.

DUNCAN P. MACPHERSON
Advertising Manager

700 So. Washington Square
Philadelphia, Pa.

See Standard Rate and Data Service for rates. Advertising
copy expected the 10th of each month.

Subscription price per year in the United States, \$3.00;
Canada and other Foreign Countries, \$3.60; payable in
advance.

DETERMINATION OF CARBON-BLACK BESIDES ANILINE-BLACK IN BLACK VARNISHES AND LACQUERS

By DR. G. ZEIDLER and DR. H. BRISKE

As a general rule, the determination of pigments in dried varnish films is less difficult than the analysis of the dried vehicle itself. This is due to the fact that the latter suffers considerable changes caused by chemical reactions during the drying process (for instance, drying oils, oil-modified alkyde resins, stoved aminoplasts or phenoplasts, etc.), so that it is often practically impossible to fully determine the original vehicle by chemical analysis.

However, in some instances the determination of pigments in varnish films meets with unexpected difficulties, for instance where it is desired to characterize more closely the black pigment by chemical analysis. This task is comparatively easy, of course, in the case of inorganic black pigments (such as iron black) which are easily determined by simple analytical methods in liquid varnishes as well as in dried films. It is only necessary in the latter case to carefully scrape off a sufficient quantity of film substance and to isolate the pigments by common inorganic analysis.

This method proves futile, however, if it is required to determine the percentage of carbon-black in presence of black coal-tar dyes, especially aniline-black. These substances are frequently employed as black pigments in varnishes, since by careful selection and proportioning of the two types of black pigment the color tints and the gloss of the films can be accurately fixed. It is also possible by this means to exactly produce the matting effects desired.

Chemical analysis of such varnish films for the determination of the black pigment contents can be built up on basis of the three following possibilities:

- 1) The varnish contains aniline-black only.
- 2) It contains carbon-black exclusively.
- 3) Both types of pigments have been employed.

We have studied this problem on basis of a few series of experiments and have developed a method of analysis yielding satisfactory indications as to the application of the three possibilities in the varnishes, or varnish films, examined.

Our investigations were somewhat handicapped by the fact that aniline-black is not the name of one single specified substance but a general term for a group of coal-tar dyes of non-uniform chemical composition, although in most instances they are all produced by more or less modified oxidation of aniline and related aromatic bases. They are chemically related to the nigrosines which are also used extensively as black pigments in varnishes and lacquers. These coal-tar dyes are sometimes employed as dye stuffs in their pure state, but more frequently in connection with an inorganic vehicle. One may then be justified in calling them "pigment-black."

As far as we know, pure coal-tar dyes are also marketed by the name of "pigment black" in spite of the fact that they have not been precipitated on a vehicle. It is evident, therefore, that the term "pigment black" does not signify that the organic coal-tar dye has been precipitated on an inorganic vehicle.

Inorganic vehicles for aniline-black or nigrosin during the process of manufacture frequently are kaolin or barium sulphate, and it can be expected, for this reason, that a perfect separation of coal-tar dyes and vehicle is easily possible even by chemical means. Even if it proves possible to remove a large part of the organic substances from the pigment by chemical operations, the remaining inorganic vehicle, or substratum, exhibits a sufficient degree of blackness to be mistaken for carbon-black. This is particularly true if aniline-black is precipitated on carbon-black, and black pigments containing special types of carbon-black are actually employed in the varnish and lacquer industries.

It is obvious that in view of these conditions, general methods of varnish analysis providing for the isolation of pigments by solvent extraction and incineration of the residues will lead to wrong conceptions; indeed, it is practically impossible in such cases to determine from the composition of the residues of incineration whether the pigments consisted of straight aniline-black or carbon-black or of mixtures of these two groups of pigments.

Procedure of the Investigation

Our investigations were carried out with a clear nitro-lacquer of the following composition:

Wasag-cotton 6a (dry)	60 parts by weight
Butanol	30 " " "
Toluol	15 " " "
Butyl acetate	100 " " "
Tricresyl phosphate	30 " " "
Intrasolvan-E	65 " " "

300 parts by weight

This lacquer was pigmented by various black pigments, using a number of commercial aniline-black pigments partly in a pure state, partly precipitated on substrata. The carbon-black composition used by us was a commercial product composed as follows:

Nitrocellulose	66.7
Dibutyl phthalate	20.0
Carbon-black	13.3
	100.0

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This composition ensured perfect distribution of the pigment particles, a matter of extreme importance to the accuracy of our experiments. We added this composition in a pure state as well as in mixture with the aniline pigments mentioned above.

The lacquers were applied to glass plates in the usual manner. After thorough drying and storing for a number of days they were scratched off. The lacquer film was treated with butyl acetate to separate the pigment from the vehicles, resins and plastifiers.

After drying the residue, 0.05 grams of the purified black pigment in a test tube was covered with 7 cubic centimetres of a 50-percent nitric acid solution. The mixture was then heated to 100 degrees Centigrade by placing the test tube into a boiling water bath for 10 minutes. This treatment caused the destruction of the coal-tar dye to such an extent that differentiation of the carbon-black became possible.

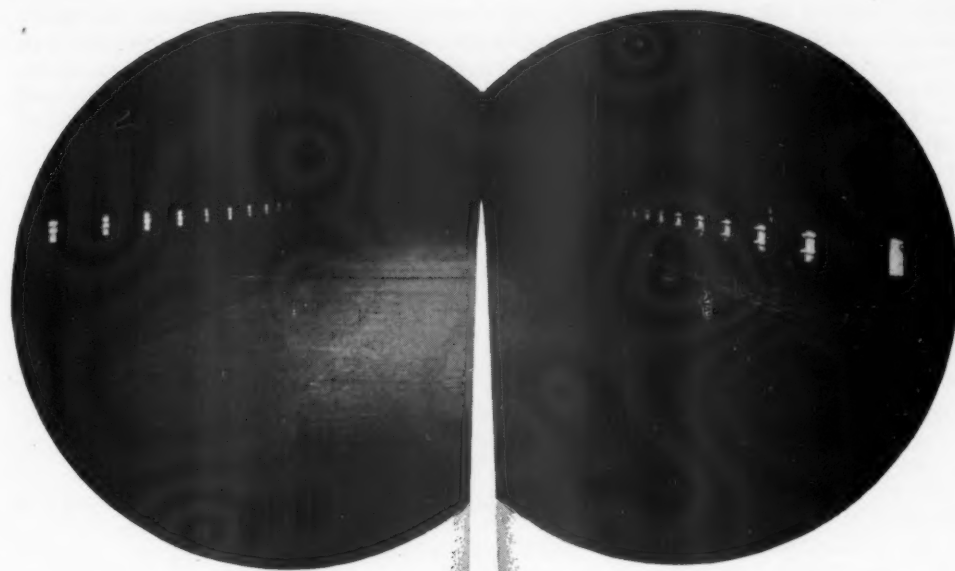
It is necessary to maintain the described concentration of the nitric acid, as more dilute solutions are not sufficiently strong while more concentrated reagents effect excessive decomposition of the mixtures. After the heating treatment the contents of the test tube are poured into a large porcelain dish and strongly diluted with water.

It is easily possible by examining the results of the nitric acid treatment to determine whether the lacquer contained aniline-black exclusively or whether it was mixed with carbon-black. Decomposition of the aniline-black is recognized by the brownish-red coloration of the solution and by brown flakes floating around in the solution and settling but slowly. If the lacquer contained carbon-black as well, this is recognized by a quickly settling black powder, i. e. carbon which is not attacked by nitric acid.

It may eventually be necessary to continue the examination if the following questions have to be cleared up:—

If the mixture in the porcelain dish does not contain a black sediment, the sample did not contain carbon-black. The matter is not entirely clear, however, if a black sediment has actually been formed. This is due to the fact that—as explained above—the last particles of organic pigments precipitated on inorganic substrata cannot be entirely removed, so that carbon-black might be indicated where in reality the sediment consists merely of kaolin colored black by residual traces of aniline-black. It is practically impossible to decompose the black coal-tar dye on the anorganic substrata to such an extent as to destroy the original black color.

If, therefore, the solution in the porcelain dish exhibits a black sediment, this can with certainty be indentified as carbon-black only if no aniline-black is present at the same time. If the solution indicates the presence of this latter substance as well, the analysis must be continued as follows:

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The black sediment is filtered off, washed and dried. A weighed portion of the dried substance is charged into a porcelain crucible and incinerated. If it is considered that carbon-black leaves practically no residue, the following conclusions are in order:

If the crucible contains no ashes the sediment actually consisted of carbon-black while if ashes are produced which on analysis yield aluminum or barium sulphate, the black pigment contained so much inorganic substrata colored by aniline-black.

TALL OIL IN PROTECTIVE COATINGS

By W. A. WACHHOLTZ, ARTHUR C. TRASK CO.

About a year ago I had the pleasure of addressing several midwestern paint and varnish production clubs on the subject of tall oil. Since that time, the supply situation on a number of critical raw materials has eased considerably. Hiding pigments are easily obtainable and Phthalic and Maleic anhydrides are in good supply. Recently the price of Glycerine has declined sharply and there are possibilities of a further decline. It is possible that other polyhydric alcohols will decline further in sympathy. Refined tall oil has recently dropped in price. The sum total of these changes since a year ago make tall oil a still more interesting raw material to the paint and varnish manufacturer.

The main source of tall oil is the southern or slash pine, yielding about 250 pounds of oil per ton of wood, or about 12%. Northern pine isn't quite as generous, producing 100 pounds of oil per ton. The type of oil obtained depends considerably on the location of the pine forest. One investigator has shown that high fatty acid tall oils are produced in the Virginia and North Carolina region and as we go farther south, the resin content increases at the expense of the fatty acid. Also, investigator, it seems to us that green while there is little seasonal change in tall oil composition according to this wood gives a higher fat content than wood which has been stored in the wood yard for a period of time. At least, we ran into some rather high viscosity oils during the winter months, which we attributed to higher rosin content.

Tall oil is a by-product of kraft paper manufacture. Pine logs about 5 ft. long and 4 to 8 in. in diameter are fed to debarking machines, along with water to aid bark and dirt removal. A conveyor then carries them to the chippers, where rotating knives produce chips about 1"x1"x1/4" thick. Next a set of screens cull out oversized chips for reprocessing. Chips visit the storage bins, then slide down into the digesters.

These digesters range from 30 ft. in height and 8 ft. in diameter to 45 ft. in height and 9 1/2 ft. in diameter. They are made of steel and take a charge consisting of 10 tons chips, 10 tons water, and 1 1/2 tons of chemicals, consisting of sodium hydroxide and sodium sulphate, which

latter is converted into sodium sulphide before it reacts with the chips. Having charged the digester, steam is turned on, the batch is cooked for 2 1/2 to 4 hours at 340° F. Naturally, a battery of digesters is used so that the flow of pulp is continuous.

In some pulp mills the vapors coming from the top of the digesters are condensed to form sulphate wood turpentine. The black sulphate liquor is separated from the pulp and sent to the tall oil recovery plant. The tall oil exists in the black liquor as the sodium soap and is often referred to as sodium resinate. In order to make crude tall oil, the black liquor is concentrated to about 25% solids by evaporation and then tanked, where the tall oil soap floats to the top of the vessel. These skimmings are carefully collected and washed repeatedly to remove any traces of black liquor. The whole is then acidulated with H₂SO₄ to liberate fatty and resin acids.

The fatty acids and rosin are then washed repeatedly to free them of residual mineral acid, tanked to settle out residual water and shipped. The Champion Paper & Fibre Company market their crude tall oil under the trade name of Trostol, which has the following analysis:

Acid Number	158
Saponification No.	165
Ash	0.179
Resin Acids	41.3
Fatty Acids (by diff.)	50.9
Unsaponifiable	7.6

Though dark in color, it finds wide use in several different industries. It can be esterified with glycerine, or with PE to yield hardeners and oil extenders. It is used by a large number of industries in a variety of ways. When converted into a glycerine, PE or sorbitol ester, it is a film former which dries slowly to a hard film.

Crude tall oil was sold commercially for several years before popular demand and the paper mills got around to marketing a refined tall oil. The earliest refined tall oils were an improvement in color, odor, and the drying characteristics over the crude material, but it was not until the late war that important strides were made towards really pale oils. Refined tall oil is produced by a variety of methods, methods mostly closely guarded by patents and other protective means.

Paper presented before the St. Louis Paint and Varnish Production Club April 12, to appear in a future issue of Official Digest.

The Arthur C. Trask Company has sold tall oil in one form or another since 1938. Three years ago a small plant was built to manufacture tall oil esters. Since then, a 1500 gallon, closed, stainless steel kettle with all the "gadgets" has been added. Like Topsy, our line of tall oil esters just grew. During those years we have found that crude and refined tall oil can and does vary. Roughly, as rosin content increases, viscosity increases and air drying improves. Conversely, as fat content increases viscosity decreases and so does air drying. Our laboratory tried to find some easy correlation between rosin content and refractive index, acid value, weight per gallon, and viscosity. There is a slight pattern, but we are still not satisfied. The surest way to insure uniform ester production is to run the above tests, and a rosin, unsaponifiable, and fatty acid content determination.

The ASTM (Modified Wolff) method for rosin content is based upon the methylation of the fatty acids and a titration of the sulphuric acid reagent and the rosin acids, employing a double end point with thymol blue indicator. Perhaps Chicago is a particularly smoky town; anyhow, we encountered a lot of trouble finding that second end point, where the murky yellow turns into a murkier blue. We substituted a pH meter with a standard calomel half cell and antimony electrode and better results were obtained. Of the series of curves, the first curve, pH versus ml KOH was very sharp, while the second curve was definitely characteristic. Fairly close checks were obtainable. We found the ASTM method, with a pH meter titration replacing the thymol blue indicator, especially useful in analyzing crude tall oils due, of course, to the color imparted by the crude oil.

There is also a modified McNicoll method, which utilizes a Napthalene Beta Sulfonic Acid in methyl alcohol solution. It involves a methylation of fatty acids followed by titration using phenol phthalein indicator. A blank is used here. However, dark tall oils gave us a very difficult time in locating the true end point.

In order to determine the unsaponifiable content, we used the ASTM method with very good results. This procedure is a saponification with KOH,

followed by an ether extraction. Close checks can be obtained. In all of the above methods, the fatty acid content is determined by difference. Therefore, it is quite important that the rosin and unsaponifiable determinations be very accurate.

The variations of some tall oils from season to season has no doubt been a headache to many of its users. Tall oil producers are conscious of this situation and are ever striving to greater uniformity. The National Southern Products Corporation have, in our estimation, done an excellent job of producing uniform tall oils. These people are not paper manufacturers, but are actual processors of tall oil. This enables them to purchase crude tall oil or soap from a number of paper manufacturers in various regions of the south. These crudes are then blended together, with the result that uniform starting materials are obtained. By carefully controlling their refining process, a very uniform and pale refined tall

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oil is produced. Colors as pale as 8—10 Hellige are obtained, and these oils appear to heat bleach to a still lighter color. The resulting esters are extremely pale.

At Chicago we produce commercial quantities of Glycerine, Corbitol, and Pentaerythritol type esters, using stainless steel esterification equipment and plenty of carbon dioxide. The process is not too difficult and with care good yields can be obtained. The time required for esterification depends, of course, upon the acid number desired. Very low acid value oils require considerably more time than oils with an acid value of say 10—15. The rosin content of the tall oil affects the speed of the reaction, with higher rosin oils taking more time to neutralize than lower rosin oils. During the esterification process, the fatty acid portion of the tall oil usually combines below 500° F, while the rosin acids combine more rapidly at 525 to 540° F.

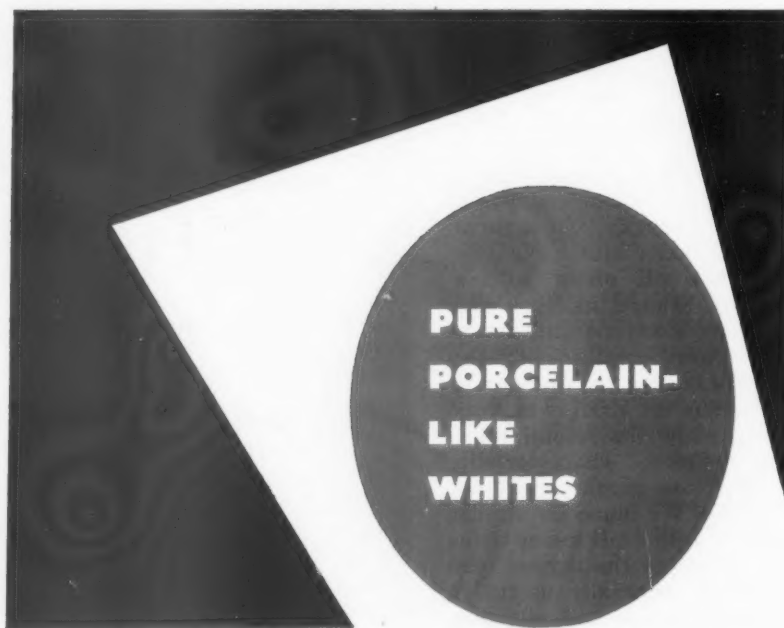
In order to obtain low acid value oils, it is advantageous to use temperatures around 540 to 550°. If glycerine esters are being made, a closed kettle with a reflux condenser is necessary, using the foregoing temperatures. It is important to reach the desired acid value in the shortest possible time, not only from a color angle and the fact that valuable equipment is tied up, but from a loss angle. Tall Oil is volatile and will distill off with the water vapor formed by the reaction and by ordinary distillation. Therefore, the longer the heating cycle, the greater the loss. Very thorough agitation is, of course, of prime importance in these esterification reactions.

Since some of the loss is due to water of esterification, losses will run as high as 10 per cent in the case of Glycerine and PE esters and higher in the case of Sorbitol esters. Maleic modifications of the foregoing esters are made without encountering too much difficulty during the process.

One of the most serious difficulties encountered in the manufacture of tallate esters is the odor and fumes. The city of Chicago has quite an extensive smoke and fume abatement program under way. In the apt words of a local wit, "You can't make a stink." The city officials are

very serious about this and are receiving the full co-operation of the Chicago Paint, Varnish & Lacquer Association and the Chicago Paint & Varnish Production Club. Both groups have a committee for the purpose of investigating this problem from an industry-wide basis. St. Louis has done an excellent job on smoke and fume abatement and if you gentlemen here tonight can give us any help on the fume angle, it would be appreciated.

Getting back to all oil fumes, these are a little more pungent and penetrating than fumes from rosin or linseed oil. It is a characteristic and clinging odor, one which is heavier than air. We have installed a system of jet injectors, wash tanks, and fume scrubbers and feel we have done a 75% efficient job. Along about the time we feel that our fume elimination equipment is doing remarkably well, some stranger will walk within 100 feet of the plant and demand indignantly, the source of "those terrific odors."



R-B-H dispersions of titanium dioxide in urea formaldehyde and melamine resins give you a chance to formulate a finish with a high, sharp, porcelain-like gloss. These dispersions are produced in an isolated manufacturing area to avoid contamination.

R-B-H white dispersions are used extensively in baking-type alkyd urea finishes for refrigerators, washing machines, stoves, kitchen furniture and similar applications where their qualities provide an advantage. They may be tinted with our dispersions in alkyd resins.

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Pigment dispersions in nitrocellulose; ethyl cellulose; urea formaldehyde; vinyl and alkyd resins; chlorinated rubber and other plastic binders.

For the paint industry we manufacture a glyceryl ester, known as Latol GP-20. This ester is pale in color, has an acid value of 10—15, and with .08% cobalt and 0.1% calcium and thinned down to varnish body it dries to a hard film overnight. It also bakes readily, forming a hard film in 1 to 1½ hours at 210° F. Since it is readily soluble in mineral spirits and compatible with a large variety of oils and resin solutions, we recommend it as a low priced extender.

It is also an interesting tool for the varnish manufacturer. It can be cooked with China Wood Oil, adding it initially, or later as a check oil. It has about the same checking or holding action as ester gum and good drying vehicles can be produced using those two ingredients as non-volatile components. When Latol GP-20 is used in cooking varnish, a liquid resin ester and a bodied oil are simultaneously added. Latol GP-20 polymerizes slowly and seems to have considerable checking action on China Wood Oil when these two are heated together. Therefore, it suggests itself wherever low viscosity is desirable or in formulations which have a tendency to gel prematurely. Below is a suggested formulation using China Wood Oil and Tall Oil Ester as the principal ingredients: 350 lbs. 6% Calcium Napthenate

350 lbs. Latol GP-20
300 lbs. China Wood Oil
1 lb. 6½% Cobalt Linoleate
8 lbs. 4% Calcium Napthenate
86 gal. Mineral Spirits

Heat Wood Oil and 90 lbs. Latol GP-20 to 525° F. Go to 570° in 8 min. Check with balance of Latol GP-20. Re-heat to 540. Cool to 500 in 45—60 min. Add Cobalt Linoleate. Cool to 450. Thin and add Calcium Napthenate.

Specifications:

Non-Volatile	50%
Body (G-H)	I—J
Color (Hellige)	10—12
Oil Length	50 gal.

The presence of Latol GP-20 in enamels improves the gloss, gives excellent pigment wetting and imparts good flow and levelling. In fact, the addition of generous amounts of GP-20 has produced considerable sagging. Our laboratory has produced, experimentally, Maleic treated tall oil esters which when thinned to 50% solids, have a good varnish type viscosity and good overnight drying. This can be done by using either a crude or a refined tall oil and makes for a low priced vehicle. Work has been done on combinations of tall oil and Epon resins. Indications are that excellent coatings can be obtained by these combinations. Great strides seem to have been made in water and alkali resistance.

Large amounts of refined tall oil are now going

into the manufacture of alkyds. Among these lines, we offer Latol 68 and 182, which are the fractionated fatty acids of tall oil. Latol 68 contains 6—8% rosin, unsaponifiable matter on the order of 2%, the balance fatty acids consisting of Oleic, Linoleic, and very small percentages of Linolenic. Latol 182 contains approximately 18—20% rosin acids, low unsaponifiable, and the balance, of course, fatty acids of the same general composition as in Latol 68. Analysis of the fatty acid portion is as follows:

	Latol 68	Latol 182
Iodine Value (Wijs)	130.8	144.8
Linoleic (Conjugated)	10.9	14.5
Linoleic (Unconjugated)	39.2	49.5
Oleic Acid	44.6	31.8
Saturated Acids (Stearic)	3.1	2.0
Unsaponifiable	2.18	2.18

This analysis is by the American Meat Institute.

Both acids are useful in the production of alkyd resins where the presence of rosin is not detrimental. Due to their very low Linolenic Acid content, the white retention of such alkyds is surprisingly good. When these Latols are esterified with PE or Sorbitol, they produce slow drying oils. They are useful in blending with refined tall oils to produce oil of known rosin content.

Chemistry of tall oil is in its infancy. There is yet much to be learned. Like any other new natural raw material, difficulties are encountered in its use and many times these new material are used in the wrong applications. However, in tall oil we have a very abundant and low priced source of fat and rosin. By its very nature, it is one of the most versatile tools the paint and varnish technician has at his command today.

For an example, with it you can make an oil containing Glycerine, Pentaerythritol and Sorbitol Esters. It would indeed be difficult to take an oil such as Soya Bean Oil and produce a similar combination without a lengthy process. It can be used in any number of combinations to form a truly wide variety of products.

Tall oil consumption in this country has increased considerably through the last several years. The soap, core oil, linoleum, paint and wetting agent manufacturers are using increasing amounts. Important strides have been made in a comparatively short time. As its secrets, its potentials and its limitations are revealed to us, still wider uses and applications will be found.

TECHNOLOGY

CONJUGATION OF LINOLEIC ACID. A brief communication concerning some tentative evidence about the total conjugation of linoleic acid upon oxidation with lipoxidase. S. Bergstrom. Nature 161, No. 4080, 55 (January 10, 1948).

FORMULATION OF ALUMINUM PAINT VEHICLES

By ROBERT I. WRAY, Aluminum Research Laboratories, Aluminum Company of America

The year just passed marked the sixtieth anniversary of the aluminum industry in America. In just about half of this period, aluminum paint has grown to its present prominence and usefulness. This rapid growth was nurtured by carefully planned and scientifically conducted research and development.

Little need be said about the era before aluminum powder was available, and some of you can remember the little cans of aluminum powder which were sold with an accompanying bottle of "banana oil" (nitro-cellulose in amyl acetate) to be used as vehicle. It was strictly a decorative product used for picture frames and gadgets. About 1920, however, Aluminum Research Laboratories set for itself the problem of making a real paint with aluminum pigment.

It was obvious that if a good protective paint was to be made with aluminum powder, a better vehicle than banana oil or gloss oil must be found. Taking a cue from the then popular raw linseed oil was first considered but it was quickly found unsuitable. The resulting paint could not be applied to a smooth vertical surface without serious running and breaking. The paint did not have a sufficiently high yield value to hold the pigment in place, and was also too slow in drying. Boiled linseed oil proved little better. Although air-bodied linseed oil proved more satisfactory, the resulting paint gave a film which was too "soft" for many applications.

Finally the so-called spar varnishes were tried and this type, considerably modified, has become the most generally accepted vehicle. The term "varnish" however, covers an extremely wide variety of products, not all of which were suitable for making aluminum paint. Furthermore, no one varnish was satisfactory for making aluminum paint suitable for all uses. Any attempt to make an all-purpose aluminum paint therefore involved a compromise in the properties of the vehicle as well as the pigment. During the past 10 to 15 years, there have been many advances in the raw materials field, and consequently many new types of varnishes have been developed. Aluminum Research Laboratories have been actively investigating these new materials and co-operating with the manufacturers in their formulation into aluminum paint vehicles.

General Characteristics of Suitable Varnish Vehicles

Varnishes which have been found suitable for aluminum paint vary from 0 to 80 gallons in oil length. The principal difference between alumi-

num paint vehicles for exterior and interior use lies in their oil length. For painting metal and other hard surfaces, 40 to 50-gallon varnishes have been found to give good service, while for painting weather exposed wood, the varnish should usually be 70 to 80 gallons in length. The longer oil varnishes are required for painting wood in order to provide the distensibility necessary to withstand such dimensional changes as may result from moisture penetrating the film and being absorbed by the cell walls. While good distensibility is also necessary in metal protective paints, high moisture resistance is also important. Varnishes of 40 to 50-gallon length possess good moisture resistance together with adequate flexibility for painting metals. For exterior use, the aluminum paint should be mixed in the proportion of two pounds of standard grade paste per gallon of vehicle.

Vehicles for aluminum paint for general use on interior surfaces need not be as long in oil as exterior vehicles. The formulation of these vehicles is not as critical in most cases as in the case of vehicles designed for exterior applications. Good leafing and good working characteristics are the chief requisites for interior finishes. The viscosity of interior vehicles is usually lower than that of exterior liquids; a viscosity corresponding to that of tube A of the Gardner Air Bubble Viscometer or even slightly lower is satisfactory. Varnishes of 8 to 12 gallons oil length make satisfactory interior vehicles.

The type of oil used in making the varnish is also important. On metal, where good moisture resistance is desired, varnishes high in tung oil are especially desirable. A mixture of tung oil with one of the softer drying oils, such as linseed oil, is generally most suitable. The oils selected should have the lowest practical acid value, and the varnish should be cooked in such a manner as to retain a low acid value. For the longer oil varnishes, a higher amount of the softer drying oil is preferred. In fact, varnishes of this type made with linseed oil only are sometimes used although the presence of smaller amounts of tung oil (up to 25 or 30 per cent) are beneficial in securing the best film properties.

It is now fairly well established that of the various metallic driers, cobalt is one of the few which does not adversely affect the leafing properties of aluminum pigments. The naphthenates and octoates appear to be the most effective cobalt driers because of their greater solubility. Recently iron naphthenate driers were investigated and it was found that they did not destroy leafing of aluminum pigments. For certain ap-

Address before the March meeting of the New York Paint and Varnish Production Club. This paper will be published in the Official Digest.—Editor.

plications, mixtures of cobalt and iron driers may therefore be desirable. Such mixtures have had no serious effect on leafing of ready-mixed aluminum paints for periods up to about one year.

Choice of thinners to be employed will depend to some extent upon the type of resin used. However, petroleum thinners with high surface tension values are generally suitable. Thinners with high surface tension usually show high kauri-butanol values. Mineral spirits with a kauri-butanol value of 40 or more has generally been found to be satisfactory, although the addition of about 10 per cent of an aromatic type solvent is sometimes beneficial. The use of V.M. and P. naphtha is not recommended because of its low surface tension.

Ester Gum Varnishes

The ester gum spar varnishes were about the first type of varnish vehicles employed for aluminum paint. Much good experience in field service has been obtained with aluminum paint made with varnishes of this type. A typical formula for a 50-gallon varnish of this type, suitable for use on weather exposed metal, is as follows:

Ester Gum	100 pounds
Tung oil	40 gallons
Linseed oil	10 gallons
Mineral spirits	54 gallons
Aromatic petroleum solvent	6 gallons
Cobalt naphthenate (6 per cent)	3.2 pounds

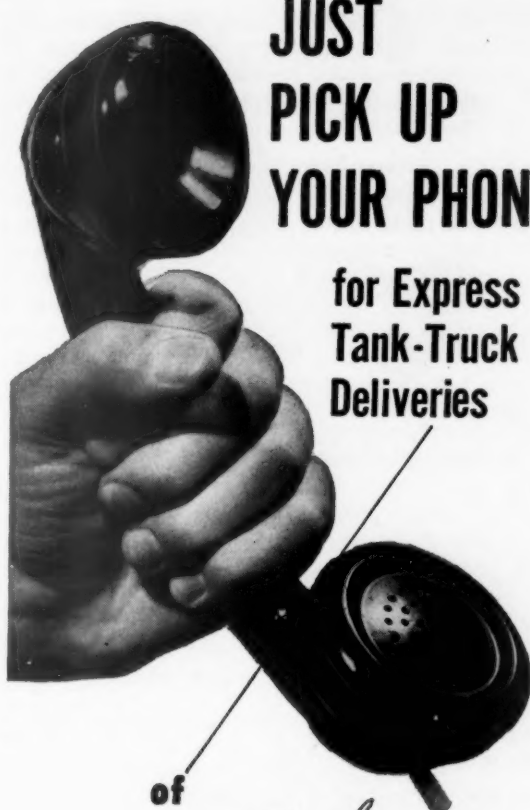
A similar varnish of 80-gallon oil length for use on weather-exposed wood is typified in the following formula:

Ester gum	100 pounds
Tung oil	16 gallons
Linseed oil	64 gallons
Mineral spirits	85 gallons
Cobalt naphthenate (6 per cent)	5.1 pounds

With proper selection of raw materials and good cooking procedure, either of the above varnishes should give excellent results in ready-mixed aluminum paint and cause no loss in leafing after extended storage.

Coumarone Resin Varnishes

Coumarone resins are particularly well adapted for use in varnishes for making aluminum paint because they are partially neutral. For this reason they have been extensively employed for this purpose. While varnishes produced with coumarone resin are generally slightly more flexible than those produced with ester gum, the same oil length varnishes are generally employed. One suggested formula¹ for use on metal consists of 100 pounds of coumarone resin to 30 gallons of tung oil, plus 20 gallons of bodied linseed thinned to about 50 per cent solids with a blend of high flash solvent naphtha and mineral spirits. Cobalt naphthenate was used as drier. An 80-gallon varnish in which the tung oil percentage was about 30 per cent was recommended for painting



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wood. Storage tests of ready-mixed aluminum paint made with coumarone resin varnishes of the above types have indicated that the paints have excellent leaf stability. They have also shown good durability on exterior exposure.

Pentaerythritol Ester Varnishes

Recently a number of the newer types of resins have been investigated to determine their suitability for use in aluminum paint vehicles. Among these are the rosin acid esters of pentaerythritol typified by the Pentalyn resins. An extensive investigation of varnishes made with these resins indicated that paints made with varnishes containing a mixture of tung and linseed oils showed better leaf stability than those made with varnishes containing soya bean and tung oils. A quick high-temperature cook appeared to be beneficial.

Very little difference was noted between Pentalyns A, F or G although varnishes made with the last two resins were somewhat more flexible, as determined by the kauri reduction test, than varnishes made with Pentalyn A. A typical formula of a varnish made with a resin of this type is as follows:

Pentalyn resin	100 pounds
Tung oil	34 gallons
Linseed oil	6 gallons

It was cooked at a top heat of 575° F. and thinned to a non-volatile content of about 53 per cent with a mixture consisting of 70-80 per cent mineral spirits and 20-30 per cent xylol. Cobalt naphthenate was added as a drier. The finished varnish had a viscosity of tube C of the Gardner Air Bubble Viscometer and passed a 75 per cent kauri reduction test. Aluminum paint made with this varnish showed no loss in leafing after storage for one year. Exterior exposure tests on steel panels indicate very good durability is obtained with the aluminum paint.

Terpene Resin Vehicles

Another group of resins which have shown good results in the formulation of aluminum paint are the terpene resins. Here again varnishes of 40 to 50-gallon lengths were found most suitable for use in aluminum paint for protecting weather exposed steel. The varnishes may be prepared by the usual cooking procedures or they may be cold cut pre-bodied oils. A typical formula for a cold cut varnish made with this type of resin is as follows:

Terpene resin	100 pounds
Bodied tung and soya bean oils	40 gallons
Mineral spirits	46.5 gallons
Cobalt naphthenate (6 per cent)	3 pounds

This varnish contained 59 per cent non-volatile matter and had a viscosity between tubes B and C of the Gardner Air Bubble Viscometer. It passed a 75 per cent kauri reduction test. The paint set to touch in two hours. As a rule, these

resins are almost neutral and when blended with oils of low acid value, the resulting varnishes have no adverse effect upon the leafing properties of aluminum pigments in ready-mixed aluminum paint. Aluminum paints of this type have given good protection to weather exposed steel in outdoor exposure tests.

Petroleum Resin Varnishes

Investigation has also been made of vehicles made from petroleum resins of the oxidizing type. About the same oil length varnishes as in the case of other resins were found desirable with these petroleum resins. Since they are very low in acid number, they give satisfactory results in ready-mixed aluminum paints. A typical formula for a vehicle suitable for use on weather-exposed metal is as follows:

Petroleum resin	100 pounds
Bodied linseed oil	40 gallons
Mineral spirits	54 gallons
Cobalt naphthenate (6 per cent)	2.7 pounds

This varnish contained 60 per cent non-volatile matter and had a viscosity of tube C. It had an acid number of 3.2 and passed a 60 per cent kauri reduction test. The paint set to touch in about three hours and dried hard overnight. It showed excellent leafing characteristics.

Phenolic Resin Varnishes

Varnishes made with pure phenolic resin are excellent vehicles for aluminum paint when properly formulated. One varnish of the general type which has proved very satisfactory in marine applications is the varnish conforming to U. S. Navy Specification 52-V-15e. It is a 25-gallon para-phenyl phenol-formaldehyde varnish containing equal parts of tung and linseed oils. When Cobalt naphthenate is used as the only drier in this varnish it shows very good leaf stability in ready-mixed aluminum paint. However, the presence of lead drier was found to be detrimental to leafing even when freshly mixed.

For general use in weather-exposed metal, phenolic resin varnishes somewhat longer in oil may be preferable. One 50-gallon phenolic resin varnish, which contained a mixture of pure phenolic resins combined with tung oil and with cobalt naphthenate drier, has shown very good results in both leaf stability and outdoor weathering tests. Somewhat greater care is required in selecting the correct phenolic resin to assure the best leafing than is necessary with some of the previously discussed resins. While the acid value of these varnishes appears to be of but little significance, the oils employed should be selected with care to insure that no increased acidity occurs from this source. The cooking of such varnishes is also of great importance.

Aluminum paint made with phenolic resin varnish vehicles shows excellent durability on weather-exposed surfaces especially in the vicinity of the seacoast. They also have good resistance to

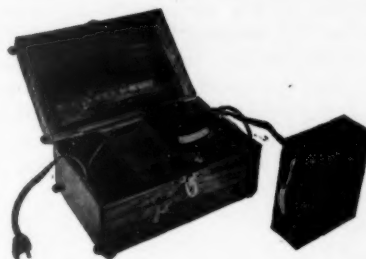
many chemical fumes. The shorter oil phenolic resin varnishes are particularly resistant to chemical attack and are widely used for this purpose. For example, aluminum paint made with 12½-gallon paraphenyl phenol-formaldehyde, tung oil varnish has given excellent protection to surfaces exposed to fumes of dilute mineral acids in a pickling room.

Alkyd Resin Varnishes

Alkyd resin vehicles show exceptionally good durability on weather-exposed surfaces. Best leafing is shown by the group of resins designated as long oil alkyds. Another requisite for good leafing properties is a low acid value. This is particularly important in the case of alkyd vehicles. High acid value alkyd vehicles cause very rapid loss of leafing of aluminum pigments. Advantage is sometimes taken of this fact in producing leaf-free aluminum polychrome finishes.

It has been found difficult to produce alkyd resin base ready-mixed aluminum paint with satisfactory leaf stability. A few satisfactory paints have been prepared using specific alkyd resins. The resins which were found satisfactory were long oil alkyds (24-32 per cent phthalic anhydride) which had acid values of about three. The kind of drying oil modifier is also probably important although this has not been fully investigated. Unless it can be established that any

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particular alkyd resin base aluminum paint has satisfactory leaf stability, it is suggested that it be used in freshly mixed condition.

Vehicles for Interior Use

The choice of oil and resin for vehicles for interior use will depend largely upon the manufacturer's preference. However, experience has indicated that the short oil coumarone resin varnishes are particularly well adapted for making this type of aluminum paint. One such vehicle which has been widely used had the following composition:

Coumarone resin	100 pounds
Bodied fish oil	12 gallons
Mineral spirits	9.3 gallons
High flash solvent naphtha	15 gallons
Cobalt naphthenate (6 per cent)	0.5 pound

The substitution of tung oil for the bodied fish oil will result in somewhat harder drying finish. Linseed or dehydrated castor oil may also be employed. In any case, oils of very low acid value should be selected.

The pigmentation of interior aluminum paints may be lower than is ordinarily recommended for exterior use. About a pound and half of standard paste per gallon of vehicle usually suffices. While the standard grade of paste is satisfactory for many applications, better metallic brilliance and smoothness is obtained through the use of the extra fine lining grades. One pound to one and a quarter pounds of extra fine lining paste per gallon of vehicle is a satisfactory proportion. The so-called "chrome finish" aluminum paints contain this grade of pigment and the vehicle is usually of the general type described above.

Miscellaneous Vehicles

While the vehicles described in the preceding sections include most of the types which are suitable for a wide variety of applications, there are many other vehicles which have been developed for specific uses. For example, where a rapid rate of air drying is required, clear lacquer vehicles may be employed. These may be of the nitro-cellulose type or may consist of a solution of thermoplastic resins in solvent. Federal Specification TT-L-58 describes a suitable nitro-cellulose lacquer vehicle. The resins most commonly employed for making lacquer vehicles of the resin solution type, are the vinyl resins and the methacrylate resins. The Solutions of 20 to 30 per cent of these resins in mixed solvents containing ketones and esters have been successfully employed with aluminum pigments.

Chlorinated rubber and synthetic rubber base lacquers also find use for certain applications. Aluminum pigments do not leaf in the accepted sense in lacquer vehicles, but develop a white frosted appearance which is quite pleasing as a product finish. Because of the low solids content of lacquer vehicles, less aluminum pigment is required to produce a satisfactory coating. Further-

more, aluminum paste pigments made with mineral spirits are not usually compatible with these vehicles because of their low tolerance for mineral spirits. It is, therefore, necessary to employ dry aluminum powder or special pastes containing aromatic solvents. With powder of standard lining fineness, 10 to 12 ounces per gallon of clear lacquer are sufficient. Even less of the extra fine lining powders may be used.

Aluminum paint used for product finishes may also be of the baking type. Many of the oleo-resinous vehicles described previously may be employed or vehicles especially designed for baking finishes may be chosen. The phenolic resin varnishes are particularly well adapted for baking finishes. These varnishes should be of moderate oil length with a high tung oil content. Alkyd varnishes which are specifically designed for baked finishes do not make particularly good vehicles for baked aluminum paint because they usually have a rather high acid value and hence do not permit good leafing. Where leafing is not important, however, they may be employed. The recently developed styrenated alkyd resin solutions have shown some very interesting results in baked aluminum finishes. These finishes have the advantage that they may be baked rather quickly at a relatively low temperature. The resulting finish is extremely hard and shows good resistance to abrasion.

Special vehicles are required for aluminum paint which must withstand high temperatures. These vehicles must be of a relatively low viscosity (about 10 to 20 centipoise) and usually have a solids content of 30 to 40 per cent. The oil content must also be low; in fact, satisfactory vehicles have been formulated which contain no oil at all. Where oil is used, fish oil is perhaps best suited because of its good resistance to high temperatures.

One of the best heat resisting vehicles consists of silicone resin dissolved in toluene². Aluminum paint made with two pounds of standard paste in a vehicle consisting of 39 per cent silicone resin in toluene showed excellent color retention and adhesion to a black iron panel after being heated to 500° C. (932° F.) for 24 hours. The silicone resin may also be blended with lower priced material and still retain some of its beneficial effect. Another excellent heat resisting vehicle was prepared by diluting a medium-long oil length (fish oil type) alkyd resin solution with an equal amount of mineral spirits. The resulting vehicle had a non-volatile content of about 35 per cent with a viscosity of between 10 and 20 centipoises. Aluminum paint made with this vehicle showed good adhesion and color retention after 48 hours heating at 500° C. (932° F.).

There are undoubtedly many special proprietary vehicles which will give excellent results for special purposes. Since the trend is definitely towards ready-mixed aluminum paint, the question of leaf retention of the mixed paint in storage

is usually important. While it has not been possible in this brief discussion to include every type of aluminum paint vehicle, most of the types have been described.

References

- (1) Kenny, J. A.—Paint, Oil & Chem. Rev. 103 No. 5, p. 20 (Feb. 27, 1941).
- (2) Houston Production Club Paper—Official Digest No. 262, p. 534 (November, 1946).

THE PRINCIPLE OF GRINDING PAINT ON A CARBORUNDUM STONE MILL*

By G. H. MOREHOUSE, Morehouse and Company

The method of stone mill grinding is not new—as you know, the old large diameter, slow speed burrstone mills were the forerunner of the modern, high production units which today can be found in thousands of paint plants all over the world.

The transformation of a premix of pigment and inerts with suitable vehicle from a pasty mass to a smooth, free flowing finished paint requires considerable work in terms of energy be the transformation obtained by the use of ball mills, by the use of roller mills, by the use of carborundum stone mills, or other methods of grinding.

* Presented before the Paint and Varnish Production Clubs: Southern in March for publication in the Official Digest of the Federation of Paint and Varnish Production Clubs and is published by their permission.

The carborundum stone mill, as we know it today, is not a development conceived and accomplished overnight, but is one in which many years of highly specialized engineering work have been expended. The units available today are small and compact, occupy very little floor space, and are readily adaptable for quick and somewhat simple installation. Their engineering features are so designed specifically with one thought in mind—to gain high production rates in proportion to the finished grind desired.

The design of the stone itself, as we know it today, proved to be a rather complicated task. The theory of grinding with such equipment requires the presence of shearing points running either in contact, or just out of contact where

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clearances are in terms of thousandths of an inch, but where the design is of such a nature that open areas exist, in a type of porosity throughout the entire stone. Were stones to be used without these open areas between shearing points—and instead the stone was cast as a solid non-porous material—you would find production would decrease rapidly, heat generated during milling would increase, and the finished grind would not be up to your standards.

In addition to the proper selection of grit size for the job involved, and the proper relative density, it is also important that stones for this equipment be so designed that as they wear, new surfaces, as presented, should be clean cutting on the stocks passing through the mill. The passage of any one particle of stock through the mill is one involving a number of revolutions in ever increasing, widening spiral paths of progress between the stone. In addition, the stock is turbulated very violently between shear points and openings between shear points. The combination of this shearing action and turbulation action brings about rapid breakdown of agglomerates, which is what you are after.

Remember that the grit size of the carborundum in the stone has a direct relation to the size of the open areas between the shear points because of the porosity in the stones as mentioned before. If a particular product does not require a fine grind, the standard 46 grit stones manufactured for paint work will give the highest production for a grind of, say up to 5, with variation of clearance between the shear points from just off of touch to contact between shear points. For finer grinding the use of 54 grit stones, with some loss in production, will bring about this result, with grinds as high as 7 plus. Some plants are using 60 grit stones in special cases.

With the production of stone of the types that are being offered today with a wide range of carborundum grit sizes being available, it is possible by the proper selection of grit sizes in the stone, by the proper consistency of the premix, and by the control of the rate of delivery of stock to the mill, to obtain finished products through carborundum stone mills at fine grinds and at high production rates.

Early models of this equipment were so designed with only one bearing and solid connectors between the motor shaft and the driving shaft, that even normal wear at either point allowed sufficient movement in the plane in which the lower revolving stone rotated to make it impossible to obtain the finer grinds which the modern, dual bearing mills of this type can now offer.

A plant using this equipment is not confined to very narrow limits of utilization in the production of paint products—premixes of very heavy consistency, or finished can volume, are readily handled. However, there are factors which will contribute to the ultimate in fineness of grind and in production capacity.

Some years ago one of the members of the Southern Production Club voiced the idea that possibly the best starting point in the utilization of carborundum stone mill equipment was to be certain that the premix being fed to the mill was of such a consistency that due to adhesion of the stock to the stone surface and with the proper amount of tack in the stock, the motor which was designed to deliver 20 horsepower would be so loaded in doing work on the unground stock, that full utilization of the motor's available power would be consumed.

We find, however, that in many instances—such as water emulsion paints—it is absolutely impossible to prepare a premix which will have a horsepower demand equal to that of the motor because of the physical character of the stock itself. Products such as straight oil or oil-resin vehicles are ground to the finest grind, will run at the coolest temperature and will give the most production when the stock being fed to the mill is of a proper consistency so that the mill will consume the available horsepower built into the motor equipment. The solids content in any vehicle may have a great effect on the action in the mill—some vehicles at certain solids content give a cushioning effect to the pigments and inerts thus not allowing for complete breaking up of agglomerates.

This same gentleman also made the suggestion that the use of high boiler solvents, such as butyl cellosolve, in quantities relatively insignificant could play a very important role in the proper processing of many products. It has been found through practical experience, not in our own technical service laboratory, but in plant of members of this group, that the addition of such products as butyl cellosolve are helpful in controlling stock going to the mill when present even in such small quantities as 1 pint to 125 gallons of premix.

Particularly, it has been found most helpful in the production of red lead, where the normal temperatures involved in stone grinding can be a detrimental factor, especially if you use linseed oil of high acid number or if you use red lead with a high litharge content, to add a small quantity of butyl cellosolve. It has also been found that the use of fine oil, where certain alkyd vesicle stocks are concerned, facilitates in a great measure the ease of handling such products through this equipment. The use of wetting agents, the use of zinc naphthenate, and the use of other additives, can be of assistance in obtaining the best production values with the highest quality.

These mills are normally equipped with a spinner in the head of the unit, which acts as a very highly efficient secondary mixer, and also as a feeding device to force premix stocks to the vortex opening between the stones. In addition, it acts to some extent as a screen thus preventing large pieces of tramp materials from entering the grinding area. The action of this spinner is, in a fashion, to pressurize the stock to the grinding area. The removal of this spinner will slow

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down the rate at which the stock is fed to a longer time. The result of this is to bring about a finished product—everything else remaining the same—of finer grind values.

We have found in our contacts with the printing ink trade that the removal of the spinner will bring about a finished ink in less time with fewer number of passes.

Tandem grinding, which may be new to you men in the Southern area, is not a new idea in many parts of the country. Many plants are running two mills—passing stock through the first, directly into the second, producing a finished product of very fine grind at high production rates. Dual passing of stock, that is twice through one mill, although similar in action, will in many cases bring about the same results. Tandem passing has the advantage of feeding the second mill with a heated stock on which 98 per cent of the work has been completed, and leaving the second mill to finish only the last remaining bit.

In many cases we find that the first mill in a tandem set-up will be operated with the spinner in place, while the second mill will be operated without the spinner. Inasmuch as these vertical mills operate without vibration, and therefore do not need to be permanently attached to the floor or to a stand, we find many mills so adapted to operate as single grind mills, or by mounting one mill on a base mounted on rubber tire rollers, with electrical plugs and water and discharge lines, a tandem set-up can be used without difficulty. We also find the portable mill mounting an advantage in that by means of a supplemental chute from a pair of twin mixers mounted normally to feed a roller mill the discharge of premix can be routed to these carborundum stone mills when the roller mill is not operating, or when a plant is grinding a stock containing abrasive materials best not run over a roller mill.

New designs of castings in the grinding area and heads of these units have shortened the length of time for complete clean up in order that colored stocks may be run. Here again we draw on our experience with the printing ink trade in that one mill may be operated on a num-

ber of different colors in any one day. The ink trade has adopted a policy of operating each mill with 6 or 7 sets of stones—one set for white, one for black, and the other five for various ranges of colors being handled.

The new spline type spindle and lower stone plate effect quick removal and clean-up of this part of the equipment and the new design in the frame and head castings allows for clean-up of these areas in a very short time with a spatula and rag with solvent.

Most user in the paint field, I am sure, have looked on carborundum stone mill equipment as fitted only to handle white goods, or have made only partial use for colors. These new changes I assure you will bring about more complete utilization of this equipment in your plant.

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of the vehicle and solvent, then adding the balance of the pigments and the necessary liquids, then running again through the mill, brings about fine grinds with over-all high production rates.

We also find that inerts are often dispersed and ground in such liquids as ester gum solution or castor oil for production of lacquers, by the use of flushed colors. These mills are also used for final mixing of lacquer stocks after premixing to level out any inequalities in such products. Again, in the printing ink field, these mills are being used to produce flatting pastes which have as a binder, nitrocellulose and consist of nitro, whitening and a solvent such as butyl acetate.

Metal primers, which so often require long ball milling, can be passed through these mills to final grind requirements, or the use of this type equipment first can bring about a reduction in the time of ball milling.

The usual speed at which these mills operate is 3600 rpm., but a recent development now makes available a unit running at 4500 rpm. and obtained by stepping up the spindle speed through the use of a gear box. Equipping this new type mill with a larger horsepower motor—40 instead of the conventional 20—brings about production capacities 3 to 4 times over those obtained by the slower mills. In one eastern plant now operating these new faster units, production rates in excess of 700 gallons per hour of house paint with a grind of 4 have been obtained. Surely, this is a long way from the old production rates obtained on burrstone mills. The latest development offered is an external hand wheel—one complete turn advances the set of stones 1/1000 of an inch—one thus doing away with the "reach in" type of adjustment of the past.

By means of this new method of adjustment, the actual measured position of the moving stone in relation to the stationary stone can now become part of your work sheet. As the stones wear, the "zero point" is very simply reset by running the spindle up to the point of touch of the stones and then adding or subtracting a measured amount of adjustment, depending on the results you wish to obtain from the mill.

To sum up, remember that this type of equipment is versatile in its operation; however, time spent in finding the best possible combination of stocks to be fed to the equipment will more than repay you in increased production and finer grade products. The proper choice of stones, (grit sizes) the right amount of solids content in the liquids in the premix, the use of those agents which have proved helpful as additives, the rate of flow of premix to the mill—all will add to the usefulness of carborundum stone mill equipment, and a more complete utilization of it in your production schedules.

RECENT FINISHES AND COATINGS. The finishes and coatings which have been of importance recently are reviewed briefly. Some information is presented about metal coatings and phosphate coatings for *metals*, plating on plastics, ceramic coatings, and a few vinyl finishes. Anon. Materials and Methods 27, No. 1, 105-106 (January, 1948).



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THE BLOOMING OF VARNISH FILMS*

By W. ESMOND WORNUM, M.C., B.Sc., A.R.C.S., F.R.I.C.

As the study of blooming is a difficult and complex one, it is important at the start to define exactly what is implied by this term. In this paper it refers specifically to the formation of an opalescent grape-like bloom on the surface of a varnish film† under the influence of moisture or high humidity.

This association of moisture or humidity with the occurrence of bloom suggests that there is some direct connection between bloom formation and the condensation of moisture upon the surface. If this view is taken, the logical approach to the subject of bloom formation is an appreciation of the fundamental condition determining condensation. In the case of varnish surfaces, then, whether or not condensation takes place under given conditions will depend primarily upon the presence of water-attractive or polar groups, in so far as these will act as condensation nuclei.

In this way the occurrence of bloom becomes directly associated with the water-attractive propensity of the surface, or more particularly with the surface polar activity. When, therefore, a condition of high humidity prevails in close proximity to a cool surface, the degree of condensation which takes place is a function of this surface polar activity and, on this account, varnishes which have different surface activities will be expected to show differences in behaviour when exposed to humid conditions.

These differences in surface polar activity have, indeed, been discussed and measured by N. K. Adam and R. S. Morrell¹ by means of contact angle determinations. As a result of their investigations they have been able to group varnishes into two broad classes, namely, those which bloom and have a contact angle below 60°, and those which are non-blooming and have a contact angle greater than 80°.

In tracing the development of bloom, then, attention must first be directed upon this question of condensation, in order to ascertain to what extent it is a primary factor in bloom formation. It is true, for example, that if moisture condenses in the form of very fine droplets or a mist upon a surface, it does give the general appearance of a bloom. Such a misting-over, however, should disappear again as soon as conditions permit evaporation of the condensed moisture, and the surface should revert to its former appearance when this has occurred. If anything other than this transient bloom or misting takes place under these conditions, it will then become necessary to divorce cause from effect and to ascertain in what manner the condensation has given rise to changes in the surface of the film to produce a more lasting result.

A careful and comprehensive study of bloom has

been made by G. F. New,² who, by means of micro-photographs, has demonstrated the formation of two different types of bloom, resulting directly from the condensation of moisture on varnish surfaces.

The first of these New has termed *Crater Bloom* owing to its character and mode of occurrence. New found that if condensation takes place on a varnish film at a critical stage in the setting-drying process, the fine droplets leave their imprint of numerous small craters on the surface. New was able to reproduce this result at will by exposing the films for a short period to warm moisture 2 to 8 hours after application, depending on the varnish. It is evident, here, that this form of bloom arises under specially adverse conditions at the time of application, but only under these conditions. From its nature, i.e. on account of the mechanical damage to the surface of the film, this bloom is of a permanent type and should be visible soon after drying has taken place.

The second form of bloom identified by New has been termed by him *Crystalline Bloom*, because this resulted from the deposition of salts (mainly ammonium sulphate) by the condensed moisture. This type of bloom, being a surface deposition, is readily removed by washing with water and is therefore of a temporary type, although after a prolonged period the crystals, dust particles, etc., may become embedded in the surface and assume a more permanent form.

Both these types of bloom can be readily recognised and their occurrence is probably fairly widespread, when conditions for their formation are favourable. It is submitted, however, that they do not give the whole story of bloom formation. For example, accepting the validity of Adam and Morrell's contact angle measurements as a criterion of surface attraction for water—and this does hold good as a broad generalisation—the degree of blooming of varnishes having contact angles of the same order may differ appreciably, particularly when blooming arises some days or even weeks after application.

This cannot be simply explained in terms of Crystalline Bloom, neither can the character of Crystalline Bloom always be identified. Furthermore, a bloom may often assume a permanent character at an early stage of its history, yet, from the manner of its occurrence, it cannot be classified as a Crater Bloom.

It is submitted, therefore, that blooming does not arise only under conditions in which actual condensation is the primary factor in bloom formation, as obtains in these two types of bloom, but that it can and does arise also from changes either chemical or physical in origin induced in the surface of the film as a result of exposure to humid conditions. For reasons, which will be seen

* Read at a meeting of the Hull Section, Oil & Chemists' Assn.

† Note—This term is, of course, equally applicable to paint films, but these are not considered in this paper.

later, it even seems probable that actual condensation of water on the surface is not necessary to produce these changes, but that they may otherwise be brought about by the agency of water vapour.

Investigations into the development and nature of blooming of this character is very much more difficult, particularly as some form of Crystalline Bloom may be superimposed. Attempts to get an insight into this type of blooming have therefore been made indirectly, by a comprehensive study and analysis of the physical and colloidal characteristics of varnishes which, at the one extreme, show a pronounced tendency to bloom and, at the other extreme, are typically non-blooming.

As a result of these experiments, which were carried out over a number of years prior to the war, certain broad observations can be made. First, it has been found that, in the case of a typical blooming varnish, e.g. a tung oil-rosin ester boat varnish, the closer the varnish is taken towards gelation in the heat treatment, the more severe is the resulting bloom. Conversely, by the addition of dispersing or peptising agents which suppress gelation, e.g. oil acids, mono-glycerides, etc., the proneness to bloom and the degree of blooming is reduced to a very marked extent. In this connection, however, it must be pointed out that such peptisation materially alters the film properties in respect of water-resistance on immersion, which leads to the second general observation.

Blooming and non-blooming varnishes differ very widely in their behaviour to water, in fact they lie at the opposite extremes. Blooming varnishes normally show good behaviour to water, whereas non-blooming varnishes show very poor resistance and whiten at an early stage, the film finally chalking badly on drying out. This observation is held to be significant.

Third, blooming varnishes usually dry quickly to a tack-free film, velvet-like to the touch, whereas non-blooming varnishes dry very much more slowly and normally exhibit a noticeable tack when pressed upon by a warm hand.

These observations, particularly the latter, draw attention to the physical and

colloidal character of these two different types of films. They suggest that in the blooming type of varnish the colloidal complexity, i.e. micelle formation and aggregation, is of an advanced nature, and that appreciable swelling has taken place, either during the heat treatment or on drying. In the case of the non-blooming varnishes the transition from the sol to the gel state is much less marked and swelling, if any, is of a much lower order.

Experiments directed to test this reasoning give interesting confirmation. For example, as already stated, in the case of the tung oil-rosin ester boat varnish, the close approach to gelation during heat treatment, whereby colloidal complexity is developed and is accompanied by swelling (is indicated by the difficulty of temperature control among other factors), tends towards increased bloom. On the other hand peptisation, which suppresses gel formation and the building up of large

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micelles and micellar aggregates and represses any swelling, decreases the proneness to bloom, but produces films which approach the non-blooming varnishes in their general behaviour as regards tack, water-resistance and rate of drying.

In view of these observations and other considerations, it appeared probable, then, that the factor of swelling, which was usually evident in blooming varnishes, might itself be a contributing cause of bloom formation, because, where there is swelling there remains the liability of syneresis, or de-swelling, at some subsequent stage. If, for example, syneresis did take place on exposure of the film, it would manifest itself as an oily exudation and the surface would either contract or show a more open structure (at an advanced stage), and these factors would present the appearance of a bloom.

Somewhat dramatic evidence in this direction was provided in the case of one tung oil-rosin ester varnish, which was taken close to gelation. After thinning, this varnish was filtered through a bed of activated charcoal, with the purpose of upsetting the swelling equilibrium. After filtration, it was observed that the varnish had separated into two phases, which became one-phase again on mixing. When this varnish was applied, it was found to dry in the normal way except that the actual surface remained wet with a thin film of oil, presumably decomposition products which had been adsorbed during the heat treatment by swelling, had been subsequently released by the filtration, and had separated out on to the surface on drying.

In this experiment, it is true, syneresis or deswelling had been forcibly induced by filtration through activated charcoal. Nevertheless it gives a clue to the part syneresis might play in bloom formation if it was brought about by some other agency, for example, water vapour, when such swollen films were exposed to humid conditions.

To understand the significance of this observation it is necessary to take into consideration the factors involved in swelling. Swelling is the result of the adsorption of a liquid phase by the

micelles or micellar aggregates, due to secondary valence activities. If these micellar complexes are formed during the heat treatment, the liquid phase associated with the swelling may be monomolecular material or decomposition products. If, on the other hand, the complexity is built up mainly during the drying of the film then, apart from any decomposition products of oxidation, solvents may also be retained in the film by swelling. Incidentally, it may be noted that the retention of solvents normally lowers the contact angle of the film.

Where an appreciable amount of solvent is held by swelling, as often obtains in quick-drying varnishes, evaporation of the solvent on ageing, which itself may lead to a reduction of gloss, will leave behind it a finely porous surface structure; some of the soluble oil phase may also be carried to the surface by the solvent and deposited there on

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evaporation. Evidence of this can sometimes be seen by breathing upon a film after intervals of ageing. Initially the condensed moisture quickly evaporates, but after a time it may be found that the condensed moisture shows up an iridescence and remains. In this case the iridescent effect is probably caused by the droplets condensing in the very fine surface capillaries, these droplets then becoming coated by a thin film of oily material lying exuded on the surface. Under the microscope the appearance is that of numerous clusters of small round globules of liquid.

It is noticeable in the case of low contact angle varnishes that moisture usually condenses in fine droplets, and these droplets, except under conditions of very high saturation, do not spread or coalesce; it is as though the film at a quite early stage is covered with a thin oily film (cf. condensation on dirty or slightly greasy plate glass). If small areas of the surface are gently washed with distilled water and then allowed to dry, however, the washed portions can often readily be identified, in some cases, days afterwards, for when moisture condenses on these areas the droplets quickly spread and coalesce to a clear film, whereas elsewhere the condensation remains in the form of fine droplets.

It seems possible in these cases that washing with water has not only cleaned the surface of exuded oily matter, but that a surface adsorption of water has taken place, at points of contact, thereby modifying the polarity of the film. It has been observed, for example, that when partially washed films of a blooming type of varnish have been exposed to conditions promoting Crystalline Bloom, this bloom was evident on the washed portions of the film, but not on the unwashed; the same bloom was evident on the uncovered areas of glass upon which the films were applied.

It must further be appreciated, however, that films are not solids in the true sense, but are colloidal systems in which some liquid continuous phase is still present. This means that when films, which have been formed in contact with air and are in equilibrium with it, are brought into direct contact with water, a change in orientation of liquid molecular substances may result, thus altering the polarity of the film at the surface of contact.

This point seems to be one of importance, particularly in regard to the measurement of contact angles, because, if the polarity of the surface of a film is altered by orientation where actual contact with water has been made, it offers an explanation of the differences which were observed by Adam and Morrell in their measurement of the advancing and receding angles. Their readings for a number of varnishes show a very considerable difference, the receding angle being very much lower than the advancing angle, as would obtain if a change in orientation occurred and the polarity of the surface of the film was increased thereby. In their paper Adam and Morrell attribute these differences to a considerable frictional effect, but

as they had no means of determining this friction they were unable to assess its true value and put forward this view as the best assumption to make to explain the observed differences.

Apart from this factor of orientation, however, there is yet another consideration to be taken into account in regard to the measurement of contact angles. This is the fact that the surface of a varnish film is not homogeneous but is heterogeneous, in that there are contiguous phases of both polar and non-polar substances. Contact angles can only refer to the "general" surface and this will be conditioned by the preponderance of the one or the other phase.

This heterogeneous condition can often be observed in the case of a high contact angle varnish by placing a drop of clean distilled water on the surface. The drop will stand up high and be clearly defined. If now the surface is breathed upon, a clear halo may be visible around the drop, on the outside of this halo the moisture condensing as a mist. The presence of this halo can be attributed to the spreading of a thin film of water over the polar areas in the immediate neighbourhood of the drop, while the influence of the non-polar groups or areas opposes the actual spreading of the drop itself.

When determining an advancing contact angle of a film, therefore, the non-polar phases opposing the spreading of the water on the surface will tend to give a high reading. When determining the receding angle, on the other hand, because the polar phases have now been pre-wetted by contact with water, a low reading is obtained. This consideration, together with the orientation factor, means that contact angles can at best only be regarded as an approximate guide and do not give exact information of the surface characteristics; they are none the less valuable in drawing a broad differentiation, particularly in regard to the pronicity of a surface to Crystalline Bloom.

1. N. K. Adam and R. S. Morrell, Paint Research Association, Technical Paper No. 2 and No. 3, 1928. Also *F.S.C.I.*, 1934, **53**, 255r.

2. G. F. New, Paint Research Association, Technical Paper No. 7, 1929.

Continued in July issue

TECHNOLOGY

PIGMENTED RESIN EMULSIONS FOR TEXTILE TREATMENTS. In a paper presented before the Cellulosic Fibers Group Meeting of the American Association of Textile Chemists and Colourists at the National Convention in Chicago, the author discussed the use of pigmented resin emulsions for printing and pad dyeing of textiles. Pigments, resins, pigment resin emulsions, water in lacquer emulsions for printing and lacquer in water emulsions for pad dyeing are reviewed. Seventeen references; discussion. M. Silverman. *Am. Dyestuff Reporter* **37**, No. 2, P44-P46 (January 26, 1948).

RECENT ADVANCES IN PAINT TECHNOLOGY

CATALYTIC ACTIVITY OF LINOLEATES AND THEIR MIXTURES IN THE DRYING OF LINSEED OIL. As the literature on the relative activities of metal linoleates shows some confusion, their activities have been redetermined. In the order of decreasing activity they are classified as follows: Co, Fe⁺³, Fe⁺², Mn, Pb, Ce, Cr, Ni, Zr, Be, Tl, Cd, UO₂, Al, Th, Ba, Ca, Mg. Various mixtures were also tried. The activity of a mixture of two metal linoleates is expressed by the formula $1/C_i/T_i + (1/T_2)$, where T_i and T_2 represent the drying times of the components. D. Pagani. *Ann. chim. applicata* **37**, 12-23 (1947) (Abstr. through Chem. Abs. **41**, 7136 (1947)).

INFRARED HEATING APPLICATIONS. Rate of drying of paints, enamels, lacquers, and the heating of metals depends on their color and composition, darker colors absorbing heat much faster than lighter ones. Gold reached 321°F. in 15 min., stainless steel reached 280°F. and chrome plate 270°F. in the same length of time. Numerous industrial and commercial applications are discussed. (complete abstr.) O. O. Crutchfield. *Elec. Light and Power* **25**, No. 2, 82-4, 86 (1947) (Abstr. through Chem. Abs. **41**, 7134 (1947)).

ORGANIC FINISHES FOR METALS. Problems associated with organic finishes for metals are classified and discussed from the standpoint of treating the coatings as engineering materials and their selection on an engineering basis. Presented here is a useful manual on organic finishes approached from the viewpoint of what is needed in the finish to aid the engineer in the intelligent selection and application of finishes. The introduction takes up the types of coatings (enamel, lacquer, varnish, paint); the selection is discussed as to the kind of metal and surface conditions, decorative considerations, protection characteristics, durability factors, processing conditions and cost (a check list of selection factors is given as a guide); the composition of organic finishes is discussed as to vehicles, drying oils, resins and pigments; undercoats are classified into primers and intermediate coats; and finish coats are reviewed under the topics: enamels, lacquers, varnishes, paints and specialty finishes. H. R. Clauser. *Materials and Methods* **26**, No. 6, 95-106 (Dec., 1947).

COATING RESINS. The application of tannin as a stabilizer for phenol-aldehyde resins, after the discovery of Cohnhoff of France is discussed. In the formation of phenol-aldehyde resins, if tannin is added to the resin in the "A" (liquid) stage, the tannin acts to stabilize the liquid state. Added to a coaltar, formaldehyde, hexamethylenetetramine, ammonia mixture, tannin maintains the mass in a homogeneous state and permits separation of a liquid mixture coating the phenol-aldehyde resin in a stabilized state. L. Winner. *Plastics (Chicago)* **7**, No. 6, 68, (Dec. 1947).

ELECTROSTATIC COATING PROCESSES.

Electrostatic detearing provides a method that automatically frees the part or product being processed of this excess material that results from drain after the dipping operation and thereby presents to the oven a uniformly coated surface which can be adequately baked to a uniform finish. Supported on, and electrically grounded by, any of the usual types of conveyors, the article is dipped or flow-coated, is passed over the usual drainboard which returns most of the excess paint to the tank, and is allowed to set sufficiently so that the flow has virtually stopped. Then it goes through a strongly attractive electric field established between the grounded article and an insulated conducting grid to which a static high voltage is applied and over which the dipped piece passes. This force is particularly strong at points or prominences where drain-off is most likely to occur. Because of this attraction, excess paint is removed from these points on the work; this leaves a smooth coating ready for a standard baking schedule. The source of the electric field is a specially designed voltage pack that produces 85,000 volts pulsating direct current from the usual 220 volts 60-cycle line. In general the time required for detearing most items runs between 15 seconds and 1 minute. In the process of electrostatic spraying, an electric field is created about the item by the 85-100,000 volts of pulsating direct current applied to the insulated electrodes at the spray position. The electrodes impart charges to the finely atomized paint particles sprayed into this field from fixed guns; this causes the particles to be attracted and deposited on the piece with minimum loss of finishing material and produces a coating of extreme uniformity. The work is generally rotated to insure uniform coating even though paint is drawn to all sides simultaneously. Arthur P. Schulze. *Org. Finishing* **7**, No. 6, 9-22 (1946); abstr. from Chem. Abstrs. **42**, 468 (1948), No. 2, January 20.

WRINKLE FINISHES FOR METALS. It is emphasized that very careful control must be exercised over many phases of production processes which vary from time to time under normal operating in order to produce uniform wrinkle finishes consistently. The author discusses the following twenty-five specific factors which can have a marked effect on the finish regardless of its type or texture, and outlines methods for controlling these variables: wet film thickness, application, base metal dimensions, difference in surface texture, base metal composition, enamel viscosity, enamel age, effect of atmosphere, application to drying time, drying temperature versus pattern depth, drying time, over atmospheres, undercoats and fillers, operator technique, air and fluid pressures, spray width, air contamination, skinning, source of supply, part design, paint agitation, oil absorption, speed of applica-

tion, application of lacquer and pattern size. E. A. Zahn. The Iron Age **161**, No. 3, 78-83 (January 15, 1948).

ASSESSMENT OF SHIPS' PAINTS USED IN THE BRITISH NAVY. This paper presents considerable material on the various types of paints used in marine work. The discussion takes up anti-corrosive paints for ships' bottoms (preparation of surface, types of protective paints, electrochemical aspect, assessment, raft trials, ship trials and laboratory tests), antifouling paints for use on ships' bottoms (antifouling measures, antifouling mechanism, practical assessment, leaching rate and chemical examination), weather-work paints (marine chalking, practical and laboratory assessments), fire-retardant paints, deck paints (interior deck-paints, weather-deck paints for use over steel), anticondensation paints (tests for color, efficiency, ease of application, neutrality of condensate, minimum combustibility, adhesion of the anticondensing agent). Photographs of many tests and testing apparatus are shown. Several pages of discussion follow. Seventeen references. C. D. Lawrence and G. E. Gale. Jour. Oil & Colour Chemists Assn. **30**, 519-556 (1947).

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FOREIGN PATENT DEVELOPMENTS

PAINT CONTAINING SILVER PARTICLES. Brit. Pat. 609,760. Metals Disintegrating Co. Applied for March 21, 1946 (applied for in U. S. May 19, 1945); accepted Oct. 6, 1948. A paint or coating composition intended for use in the coating of ceramics such as glass, mica, porcelain etc. with a continuous, electrically conducting coating of silver consists of a suitable hydrocarbon vehicle containing metallic silver particles coated with a silver halide, silver tellurate, silver pyrophosphate or silver sulfate.

ORGANO-SILICON COATING COMPOSITIONS. Brit. Pat. 610,152. E. L. Warrick (assigned to Corning Glass Works). Applied for March 28, 1946 (applied for in U. S. July 25, 1945); accepted Oct. 12, 1948. A process for producing organo-silicon coatings consists of mixing an organo-polysiloxane (i.e. a silicone) with a heat-resistant filler (e.g. asbestos, silica, clay, barium titanate, fiber glass, metal oxides, slate, mica) to form a smooth, homogeneous paste, adding a minor amount of a diacyl peroxide (e.g. benzoyl peroxide) as a curing agent, and a suitable pigment. This paste is then used to coat an article, the coated article then being raised to a temperature of 180-400°C. in at least two minutes and then held in this temperature range until curing takes place. The coatings are described as tack-free, non-porous, heat-resistant, oil-resistant, water-resistant, flexible, resilient and electrically insulating, and adapted for use as coatings for metallic conductors, linings for tanks, and as protective finishes for siliceous as well as metallic surfaces.

METHOD FOR COATING SURFACES. Brit. Pat. 611,399. F. A. Rivett and T. P. Hoar (assigned to Schori Metallising Process, Ltd.). Applied for April 30, 1946; accepted Oct. 28, 1948. A process for coating metal surfaces consists of spraying from a pistol by means of compressed air, a natural or synthetic wax, a grease or petroleum jelly, in the presence of a corrosion inhibitor such as an oil-soluble sodium or calcium salt of a petroleum sulfonic acid.

FINISHING MATERIAL FOR RUBBER GOODS. Brit. Pat. 611,421. W. H. Swire and A. Hardy (assigned to B. B. Chemical Co., Ltd.). Applied for April 15, 1946; accepted Oct. 29, 1948. Surfaces of rubber or other flexible material are lacquered, painted or finished by covering them with a coating material containing a copolymer of butadiene and acrylonitrile (e.g. "Hycar O. R."), a phenol-aldehyde type synthetic resin (e.g. "Bakelites"), a suitable organic solvent and conventional pigments.

METHOD FOR HARDENING RESINOUS COATINGS. Brit. Pat. 612,762. L. H. Griffiths and P. A. Chennell (assigned to Semtex, Ltd.). Applied for June 1, 1946; accepted Nov. 17, 1948.

A process for coating surfaces with resinous compositions which harden *in situ* involves spraying on the surface an aqueous syrup of an incompletely condensed hardenable resin (e.g. a urea-formaldehyde or phenol-formaldehyde synthetic resin) and simultaneously applying a gaseous hardening agent for the syrup (e.g. hydrogen chloride gas or gaseous sulfur dioxide) to the surfaces being sprayed.

PRODUCTS USEFUL AS DRYING OILS. Brit. Pat. 613,263. J. W. Kroeger (assigned to Fred'k H. Levey Co.). Applied for June 13, 1946 (applied for in U. S. Sept. 18, 1945); accepted Nov. 24, 1948. In order to utilize cheap natural drying oil fatty acids, improve semi-drying or non-drying oils, and to modify natural drying oils, ketone-aldehyde condensation products (e.g. acetone-formaldehyde condensates) are esterified with natural fatty acid mixtures by heating the reactants in the presence of a catalyst, or heating at 500-600°F. without a catalyst. The reactants can also be combined by esterolysis in the presence of a suitable catalyst such as sodium methoxide, litharge, sodium hydroxide, zinc oxide etc. Suitable oils include linseed, oiticica, soya, castor, China wood and other glyceride oils and the fatty acids derived therefrom.

ANTICORROSIVE PAINT. Belgian Pat. 470,-240. L. N. J. H. Elias. 1947. An anticorrosive paint for iron, cement or other structural materials contains at least one rubbery constituent and at least one plasticizer (resin) dissolved in a solvent such as benzene, trichloroethylene or the like. (Abstract from Chem. Abs. 43, 1200 (1949)).

PRODUCTION OF OXIDES OF TITANIUM AND ZIRCONIUM. Swiss Pat. 221,309. H. Preis. 1942. The vapors of titanium or zirconium chlorides are heated to about 800° and mixed with preheated air or oxygen to produce the oxides of titanium or zirconium with a particle size range of 0.4 to 1 μ which may be used without further grinding or sizing as pigments. (Abstract from Chem. Abs. 43, 421-2 (1949)).

CELLULOSE ESTER AND MONOARYLUREA/FORMALDEHYDE CONDENSATION PRODUCT MIXTURE. French Pat. 878,117. I. G. Farbenindustrie Aktiengesellschaft. Applied for July 7, 1941 (applied for in Germany Feb. 28, 1940); delivre Sep. 28, 1942. Cellulose esters (e.g. cellulose acetate) and resins which are compatible therewith such as those formed by the reaction of formaldehyde with an arylurea (e.g. phenylurea) and optionally also with a smaller amount of a formaldehyde/arylsulfamide (e.g. benzenesulfamide or toluenesulfamide) are heated together to form lacquers etc. The resins are resistant to light and water and improve the adhesion of the cellulose ester coatings.

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